Fabrication and Testing of Activated Alumina Based Defluoridation Filters

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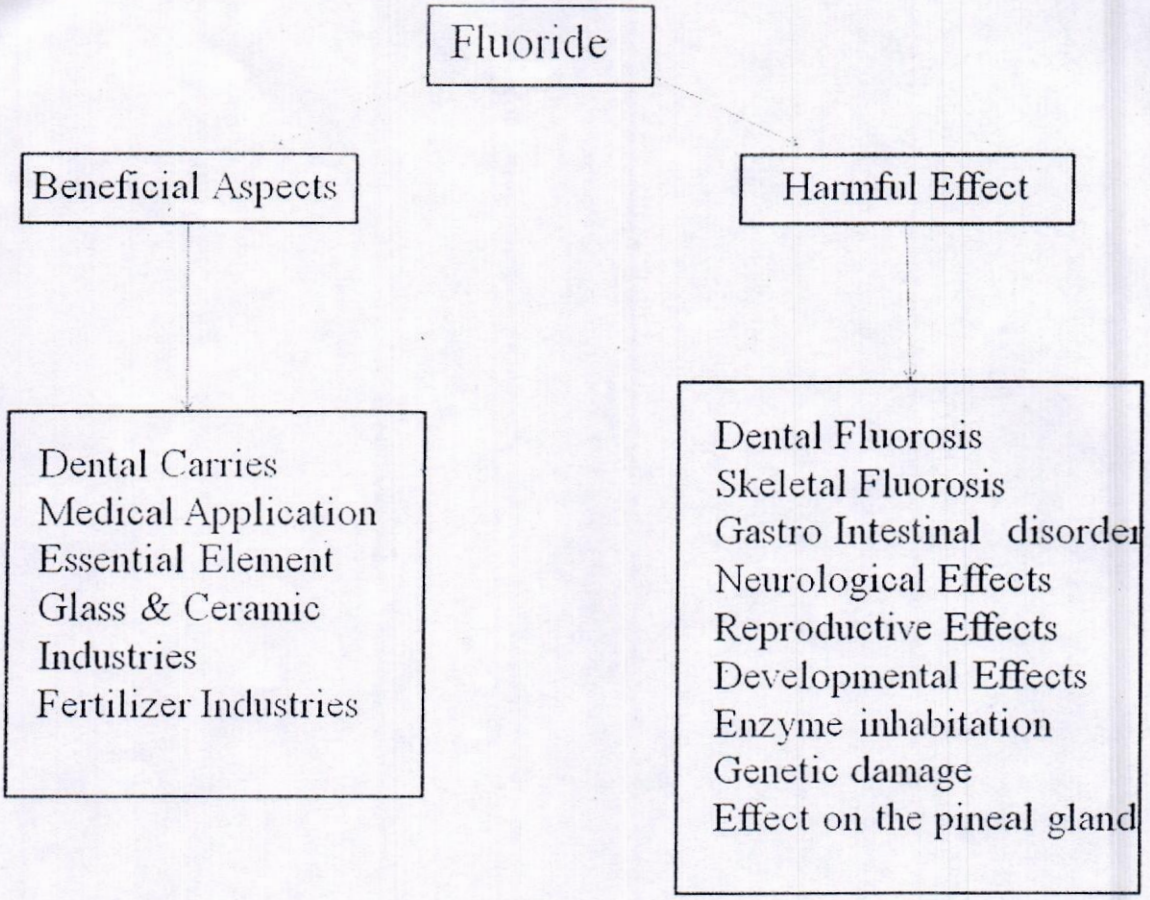
1. INTRODUCTION

Fluorine belongs to the VII group of 2nd period of the periodic table. It is the lightest member of the halogen family. Fluorine is rarely or never found free in the nature in elemental form. It is highly reactive and has strong affinity to combine with other elements.[1]

The problem associated with the excess fluoride in drinking water is highly endemic and widespread in countries like India, Pakistan, Bangladesh, Argentina, United State of America, Morocco, New Zealand, Thailand etc [2]. The problem has reached alarming proportion affecting at 17 states in India.[3] In Rajasthan, all the 32 districts have been declared as fluorosis prone areas. The worst affected districts are Nagaur, Jaipur, Sikar, Jodhpur, Barmer, Jhunjhunu, Churu, Bikaner, Ganganagar etc [4, 5, 6].

Fluoride in the drinking water of Rajasthan have been found to originate from igneous rocks, which extend from Delhi to Gujarat [7]. As the fluoride minerals are nearly insoluble in water, hence fluoride is present in ground water only when the conditions favor the dissolution of the fluoride bearing minerals. The main source of fluoride in groundwater is basically from the mafic minerals: Flurospar [CaF_2], Cryolite [Na_3AlF_6], Fluoroapatite [$Ca_5(PO_4)_3F$] [1, 8].
Fluoride in drinking water may by beneficial or detrimental depending on its concentration and total amount ingested. Fluoride is beneficial especially to young children below eight years of age when present in permissible limit 1-1.5 ppm for calcification of dental enamel [5, 9]. Fluoride has been used in the treatment of osteoporosis though beneficial effects have been reported, the dose-response relationships and efficacy need further clarification. Fluoride containing compounds are used to increase the fluidity of metals and sludge in the glass and ceramic industries [1, 2]. Anhydrous hydrogen fluoride is used in the production of most fluorine-containing chemicals and in the production of refrigerants, herbicides, pharmaceuticals, high octane gasoline, aluminum, plastics, electrical components, and fluorescent light bulbs [10]. Aqueous hydrofluoric acid is used in stainless steel pickling, glass etching, and metal coatings. Volcanoes are the major natural source of hydrogen fluoride. Sodium fluoride has been used as an insecticide, rodenticide, and fungicide [1].



Many methods are used to remove fluoride from drinking water like bone charcoal [2, 11], contact precipitation [7, 12], Nalgonda technique [7, 13], activated carbon [14], ion-exchanger [7, 15], reverse osmosis [7], fly ash [16], electro-coagulation method [7], clay column [2, 11], activated alumina [7, 17, 18, 19, 20, 21, 22].

2. PURPOSE OF STUDY

The presence of fluoride above permissible limits in drinking water poses serious long term health problem like premature aging, inability to do physical work, impairment of nervous system, pains in joints, weakening of muscles and bones etc. Recent studies have shown that there are high levels of fluoride in ground water at many places in the Sikar district, of Rajasthan [4]. The people of the region age quickly, have mottled teeth and suffer from musculoskeletal disorders. There is no defluoridation of drinking water in this region. This is mainly due to lack of awareness of the fluoride problem among the local population.

According to Rajasthan Development Report, compiled by the Planning Commission, Government of India all districts of Rajasthan are affected by fluoride [23, 24, 25]. In Sikar, deposits of calcium fluoroapatite, fluorspars are abundant. Ground water thus becomes naturally contaminated by fluoride on contact.

3. LITERATURE SURVEY

The fluoride ion plays an important role in human physiology. High fluoride level in drinking water has become a critical health hazard of this century. In many parts of the world, high concentration of fluoride occurs naturally in ground water and causes wide spread fluorosis, a disease which mainly affects the bones and teeth [26, 27, 28]. Low fluoride concentration stabilize the skeleton system by increasing the size of the apatite crystal and reducing their solubility. About 95% of fluoride ingested the body is deposited in hard tissues like bones and teeth weaking their structure and making them brittle. Fluoride ingested with water goes on accumulating in bones up to age of 55 years. At high doses fluoride can interfere with carbohydrates, lipid protein, vitamin, enzyme and mineral metabolism [1, 3, 4].

Diseases due to Fluoride

Long term consumption of water containing greater than 1 ppm of fluoride leads to dental fluorosis. Yellow glistening patches on the teeth are seen which may eventually turn brown, and presents itself as horizontal streaks. The brown streaks may turn black and affect the whole tooth which may get pitted, perforated and chipped off at the final stage. Dental fluorosis not only poses cosmetic problems but has serious aspects too, in terms of matrimonial problems [1, 19, 21].

1. Skeletal fluorosis [1, 20]

This has been observed in persons when water containing more than 3-6 mg/L of fluoride is ingested. Skeletal fluorosis affects young and old alike. Fluoride can also damage the foetus- if the mother consumes water and food, with a high concentration of fluoride during pregnancy/breast feeding, infant mortality due to calcification of blood vessels can also occur. Some symptoms of skeletal fluorosis are severe pain in the backbone, severe pain in the joints, severe pain in the hip region, stiffness of the backbone, immobile /stiff joints, increased density of bones, besides calcification of ligaments, construction of vertebral canal and inter vertebral foramen-pressure on nerves and paralysis.

2. Non-Skeletal Manifestations [1, 3, 29, 30]

This aspect of fluorosis is often overlooked because of the misconception prevailing that fluoride will only affect bone and teeth. Flouride, when consumed in excess can cause several ailments besides skeletal and dental fluorosis such as:

1. **Neurological manifestations**:

Nervousness, depression, tingling sensation in fingers and toes, excessive thrust, tendency to urinate frequently (polydypsia and poly urea are controlled by brain-appears to be adversely affected).

2. **Muscular manifestations**

Muscle weakness, stiffness, pain in the muscle and loss of muscle power.

3. **Allergic manifestations**

Very painful skin rashes, which are perivascular inflammation—present in women and children appear as pinkish, red or bluish red spots on the skin that fade and clear up in 7- 10 days, they are round or oval in shape.

4. **Gastro intestinal problems**

Acute abdominal pain, diarrhea, constipation, blood in stools, bloated feeling (gas), tenderness in stomach, feeling of nausea and mouth sores.

5. **Kidney**

Urine may be much less in volume, yellow red in colour, itching in the region of axilla.

6. **Head-ache**

7. **Loss of teeth** (edentate) at an early age

Techniques of fluoride removal

There are several techniques of fluoride removal from water. The most prominent among them are explained below:

1) Bone Charcoal Method [2, 11]

Bone charcoal is a blackish, porous, granular material. The major components of bone charcoal are calcium phosphate 57-80 %, calcium carbonate 6-10%, activated carbon 7-10 %. Bone charcoal has ability to absorb a wide range of pollutant such as colour, taste and odour components and has a ability to take up fluoride from water. Chemical composition, mainly as hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$ where one or both the hydroxyl-group can be replaced with fluoride.

$Ca_{10}(PO_4)_6(OH)_2 + 2F^- \rightarrow Ca_{10}(PO_4)_6F_2 + 2OH^-$

The method has the following advantages: high sorption capacity, locally available, cheap raw materials (animals bones), fast reaction time (less than 30 min), no addition chemicals are needed for the treatment process; filtered water is neutral in taste and colour.

The method has the following disadvantages: limited acceptability of animals bones as filter materials for drinking water, initial investments and experience needed to set up bone char production, maintenance and regular monitoring necessary- if filter is saturated it needs to be regenerated or replaced.

The bone charcoal method of defluoridation has been set up in Thailand and Africa.

2) Contact Precipitation [7, 13]

Contact Precipitation is a technique by which fluoride is removed from the water through addition of calcium and phosphate compounds and bringing the water in contact with a saturated bone charcoal medium. This method is slow and time assuming.

The method has the following advantages: enhanced fluoride uptake capacity and filter life, locally available materials, local production and low cost.

The method has the following disadvantages: the calcium phosphate pellets cannot be regenerated; chemicals processes are not fully understood yet.

Contact precipitation method of defluoridation has been set up in Tanzania and Kenya.

3) Nalgonda Technique [7, 13]

This method uses developed in India by the National Environmental Engineering Research Institute (NEERI) and developed to be used at both community and household level. The process involves addition of aluminium sulphate and lime. The fluoride removal process is by a process of coagulation/ flocculation and sedimentation. The addition of lime ensures an optimal removal pH of around 6-7 which allows complete precipitation of aluminium. The addition of lime also helps to form dense flocs for rapid settling.

The reaction involved in this process are:

1. Aluminium dissolution

$$Al_2(SO_4)_3.18H_2O \rightarrow 2\,Al^{3+} + 3SO_4^{2-} + 18\,H_2O$$

2. Aluminium precipitation

$$2Al^{3+} + 6H_2O \rightarrow 2Al(OH)_3 + 6H^+$$

3. Co-precipitation

$$F^- + 2Al(OH)_3 \rightarrow \text{Al-F complex + undefined product}$$

4. pH adjustment

$$6Ca(OH)_2 + 12H^+ \rightarrow 6Ca^{2+} + 18\,H_2O$$

The Nalgonda process was earlier practiced in India and Tanzania.

The method has the following limitations : there is risk of contamination of the treated water by aluminium in case of overdosing of aluminium sulphate. The WHO standard concerning aluminium is 0.2 mg/l, which is very low. Also this is a work-intensive method, especially at a household level due to the need of daily mixing of alum and lime.

The method has the following advantages: Chemicals are cheap and generally locally available, treatment is well accepted by locals, and removal processes are well understood.

The method has the following disadvantages: fluoride elimination rate is low (70-80%), the right amount of chemicals need to be added, residual aluminium in the water is a constant danger, high sulphate concentration in the water make it taste salty, and daily maintenance effort is very high.

d) Clay Column [2, 11]

Clay is an earthy sedimentary material composed mainly of fine particles of hydrous aluminium silicate and other minerals and impurities. Clay is fine textured, plastic when moist, retain its shape when dried and sinters hard when fired. These properties are utilized in manufacture of pottery, brick and tile. Both clay powder and fired clay are capable of sorption of fluoride as well as other pollutants from water. The ability of clay to clarify turbid water is well known. This property is believed to have been known and utilized at domestic level to filter water in ancient Egypt.

The method has the following advantages: low cost and locally available

The method has the following disadvantages: low fluoride adsorption capacity, slow and hygienic aspects because use of clay.

e) Activated Alumina [7, 17, 18, 19, 20, 21, 22]

Activated alumina is a granular, highly porous material, synthesed from aluminium tri hydrate [$Al(OH)_3$]. It has a amphoteric character. It is a good adsorbent due to its small size, and large surface area. The fluoride uptake capacity of activated alumina depends on the specific grade of activated alumina, its particle size and the water chemistry (pH, alkalinity and fluoride concentration).

Advantages of Defluoridation Using Activated Alumina

The main advantages of activated alumina (AA) based defluoridation are as follow : It requires minimum contact time with fluoride contaminated water for defluoridation, it can be regenerated easily, percentage of regeneration is considerably high, there is very little attritional loss (to a negligible extent) during the regeneration at the initial stage of operation, it is indigenously available and cheap, defluoridation capacity at neutral pH is appreciable, although it has greater defluoridation efficiency at pH 5 -7, its defluoridation capacity is independent of temperature, AA is easy to handle, filter fabrication is easy and most importantly AA and the filter requires no maintenance and no electricity, AA filters are suitable for use in rural communities.

The effect of other ions present in drinking water like chlorides, sulphate and carbonates, over defluoridation efficiency of AA is minimum. The presence of bicarbonate ions show considerable influence in the process of defluoridation.

Mechanism of fluoride removed by Activated alumina

AA surface is amphoteric in nature and can exist as $AlOH_2^+$, AlOH and AlO^-. Fluoride binding to AA is proposed to be due to exchange of surface hydroxyl groups, which can be represented by the following reaction.

$$AlOH_2^+ + F^- \rightarrow AlF + H_2O$$

$$AlOH + F^- \rightarrow AlF + OH^-$$

Why we have chosen activated alumina technique

Bone charcoal method would not be acceptable socially in this locality because of religious beliefs. Clay is not available in the district as it is semi desert region with sand all around. Clay has to be fired which required use of fuel wood which is in very short supply.

Contact precipitation requires adding dose of chemicals and use of a bone charcoal column. The filter therefore would daily attention and bone charcoal cannot be used because of local religious beliefs. Moreover the chemicals, Ca^{2+} salts and phosphate salts have to dissolved in water before adding and stock solution of each chemicals has to be maintained.

The Nalgonda Technique is not suitable as a calculated quantity of alum and lime has to be added every day to water. It has been shown that aluminium remains in filtered water. Intake of aluminium causes Alzhimers disease.

The calcium hydroxide- magnesium oxide sodium-bisulphate method is also unsuitable as it requires adding daily dose of calcium hydroxide, magnesium sulphate and sodium bisulphate in calculated quantities. The period takes a long time 16 hr for operation and the fluoride congaing sludge at the bottom has to be carefully separated from the fluoride free top layer of water.

The activated alumina technique is the simplest and easy with two bucket system, one for storing raw water other for storing filtered water. The filtered body is fabricated food-grade, plastic or stainless steel. Activated alumina is solid and it can be handled easily also. It is available in bulk quantities and is relatively inexpensive. The activated alumina can be regenerated by using NaOH and H_2SO_4 quite conveniently. The importance of the process is that it is easy and requires minimal involvement of technical personnel is harmless and is cost effective.

Therefore we will use Activated alumina (Al_2O_3) technique for removing fluoride in drinking water.

4. PROPOSED WORK

In the proposed research work defluoridation filters of different design and capacities will be fabricated. We will use of the shelf components for fabrication. Their field trail in the village (Bhooma Chota) in Lakshmangarh Tehsil will be conducted. Investigation into regeneration process and conversion of the waste sludge into building materials will also be carried out.

5. METHODOLOGY

1. Collection of water Sample

Initial field survey to collect water samples to measure the concentration of fluoride and other water quality parameters.

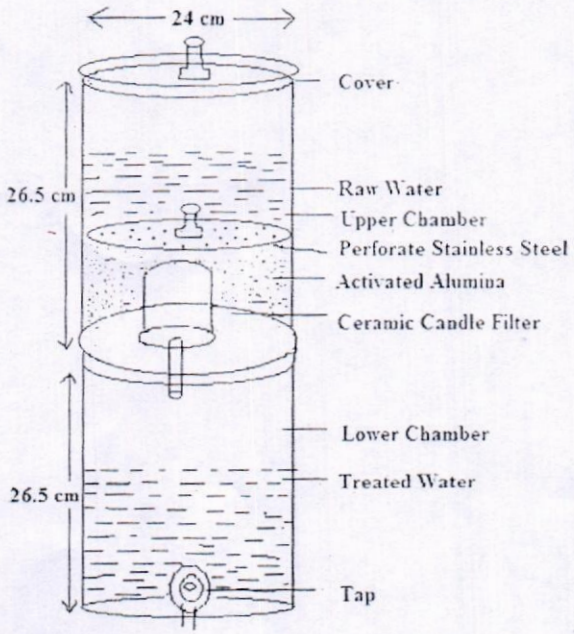
2. Study of fluoride uptake from fluoride contaminated water by AA

The time profile of percentage removal of fluoride by AA from ground water would be determined. The kinetics of the fluoride removal will be studied.

3. Fabrication of Different Activated Alumina (AA) Based filter

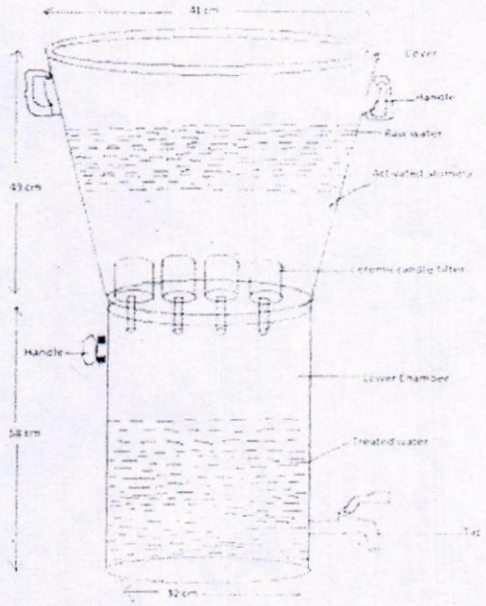
1. Fabrication of 11 litre capacity filters with ceramic cartridge: -

Foodgrade polypropylene containers of 11 liter capacity (Bharati Houseware) will be used to make the body of the filter. Ceramic cartridge will be act as flow controller and filter for suspended impurities. Plastic taps, perforated plates will be purchased locally. AA (DF-101) will be purchased from Bhargava Industries, Surat, Gujrat, India. The top container will be filled with activated alumina (DF-101, M/s Siddharth Industries, Surat, Gujrat, India) in various quantities (2.5, 3.0, 3.5 kg). Filter parameter with different quantities of AA will be determined: flow rate, volume of safe water, cost of filter, empty bed contact time, bed volume, fluoride uptake capacity, time before 1st regeneration.



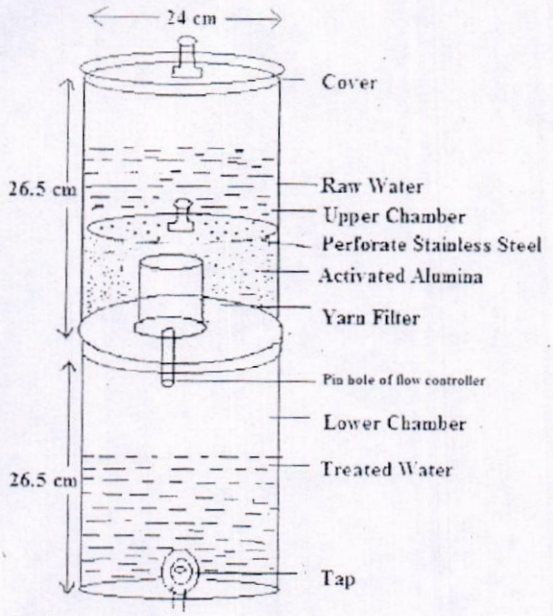
2. **Fabrication of 50 litre capacity filters with ceramic cartridge :**

Capacity of the 11 liter container will be increased to 50 liter and number of ceramic cartridge from one to four for increasing flow rate. The following filter parameter will be determined: flow rate, volume of safe water, cost of filter, empty bed contact time, bed volume, fluoride uptake capacity, time before 1st regeneration.



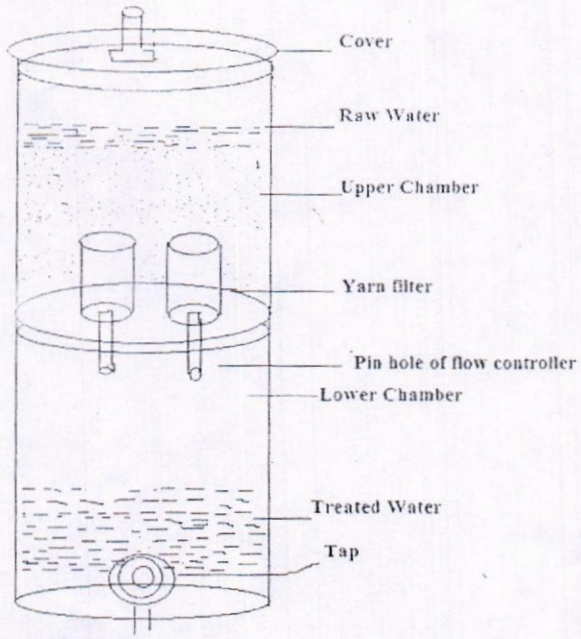
3. **Fabrication of 11 litre capacity filters with yarn cartridge :**

Foodgrade polypropylene containers of 11 liter capacity (Bharati Houseware) will be used to make the body of the filter. One yarn cartridge for suspended impurities would be used. Pin holes on the outlet would be used as flow controller. Plastic taps, perforated plates will be purchased locally. AA (DF-101) will purchased from Bhargava Industries, Surat, Gujrat, India. The top container would be filled with 3.0 kg AA. The following filter parameter will be determined: flow rate, volume of safe water, cost of filter, empty bed contact time, bed volume, fluoride uptake capacity, time before 1st regeneration.



4. Fabrication of 50 litre capacity filters with yarn cartridge:

Capacity of the 11 liter will be increased to 50 liter and number of yarn cartridges from one to two for increasing flow rate. Flow rate would be adjusted accordingly. The following filters parameter will be determined: flow rate, volume of safe water, cost of filter, empty bed contact time, bed volume, fluoride uptake capacity, time before 1st regeneration.



5. Testing of the filter

(i) Lab Testing

Water from village Bhooma Chota will be brought using a 5000 liter capacity tanker attached to a tractor. Water will transferred to 500 and 1000 liter drums in the laboratory using a pump. Further the 50 liter drums fitted with taps would be positioned on top of the filter. Short plastic pipes will channel the flow of water from taps to the upper chamber of the fluoride filter. 50 litre plastic drums will be placed below the filter to collect the defluoridated water.

(ii) Field Testing

Five filters will be installed in village (Bhooma Chotta) for field trials know to the operational problems under actual user condition.

6. Analysis of Fluoride contaminated and filtered water

Fluoride will be measured using a Fluoride Ion Selective Electrode (Orion Thermo Scientific). pH and TDS will be determined by pH and TDS meter. Sodium and Potassium will be determined by Flame Photo Meter (ESICO). Calcium and Magnesium hardness will be measured by EDTA Method and Total Hardness will be measured the addition of Ca^{+2} and Mg^{+2} hardness. Carbonate and Bicarbonate will be titrated with standardized H_2SO_4. Chloride will be determined titrate with silver nitrate and Sulphate will be measured by Colorimeter. Nitrate will be determined by UV-Spectrophotometer.

7. Regeneration of Spent Activated Alumina

a) Fabrication of Regeneration bucket

After exhaustion of AA, its needs to be regenerated. For the regeneration purpose, regeneration bucket will be fabricated. Two buckets and locally designed flow controller will be used for fabrication.

b) Regeneration Process

For the regeneration process of AA, sulphuric acid and sodium hydroxide will be used together with a nylon bag of 0.16 mm mesh size.

c) No. of Reuse Cycles of AA

After exhaustion of AA, it will be regenerated as above and reused till it is completely exhausted . The process will be repeated several times to determine the maximum practicable number of regeneration process.

8. **Sludge conversion to building blocks**

After regeneration process, sludge will obtained. It will be used for making bricks. Sludge will mixed with clay, cement and water and converted into building blocks. Its compressive strength and fluoride leaching from the building blocks will be tested.

6. Work Already Done

Major Part of the work is complete. Studies fluoride leaching from fluoride bricks remains, writing of research papers and compilation of result are in progress.

7. RESULTS OBTAINED

Paper Published in Journals

1. **R. Agrawal**, M.K. Mishra, K. Margandan, K.Singh, R. Acharya, S. Sharma, K.Qanungo, Fluoride Removal Using Activated Alumina: A Case Study of Bhooma Chota Water, Southern Brazilian Journal of Chemistry, Vol.21, No. 21, 2013.

2. **R. Agrawal**, K. Margandan, K.Singh, R. Acharya, S. Sharma, K.Qanungo, Fabrication and Testing of Intermediate Level Activated Alumina Based Defluoridation Filters, Periodico Tche Quimica, Vol.10, No. 19, 2013.

3. **R. Agrawal**, K. Margandan, K.Singh, R. Acharya, S. Sharma, K.Qanungo, Improvement of User Friendliness of a Simple Domestic Defluoridation Unit Using Activated Alumina, World Journal of Applied Environmental Chemistry, Vol. 2, Issue 1, 2013.

4. **R. Agrawal**, K. Margandan, K.Singh, R. Acharya, S. Sharma, K.Qanungo, Fabrication and Testing of a Low Cost Activated Alumina Based Domestic Defluoridation Unit Using a Ceramic Cartridge. (Communicated)

8. Conclusion

A low cost domestic fluoride filter which is easy to fabricate will be product of my research. Its various components are available in the local market except AA which is indigenously manufactured in India (Gujrat). The filter can operate with no electricity. It requires no maintenance until the fluoride in the outlet water reaches 1.5 ppm. It is suitable for rural use. The filter will be tested in lab, modified suitability and put to field trails.

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